



Research Article**Utilization of Neem Seed Oil as Surfactant in the Production of Flexible and Rigid Polyurethane Foam**

Habib Abba Sanda¹ , Muhammad Abbagoni Abubakar^{1,*} , Abdulhalim Musa Abubakar² , Mamman Bashir¹ ,
Martin Stojchevski³ 

¹ Department of Chemical Engineering, Faculty of Engineering, University of Maiduguri, Maiduguri, 600230, Nigeria

² Department of Chemical Engineering, Faculty of Engineering, Modibbo Adama University, Yola, 640261, Nigeria

³ Department of Chemical and Control Engineering, Faculty of Technology and Metallurgy, Ss. Cyril and Methodius University in Skopje, Skopje, 1000, North Macedonia

*Corresponding Author: Muhammad Abbagoni Abubakar, E-mail: magoni@unimaid.edu.ng

Article Info	Abstract
Article History	Extraction and processing of polyether polyols derived from petrochemicals, commonly used as surfactants during polyurethane foam (PUF) production, contribute to carbon emissions and raises the issue of long-term sustainability given that petrochemicals are non-renewable resources. Here, 5 mg and 4 mg of neem seed oil are employed to form flexible and rigid PUF, classified purposefully based on their divergent usage. To find an environmentally friendly replacement, flexible PUF whose mass, volume, density, compression, tensile strength, cream time, foam rise and rising time are 0.0047 kg, $2.8457 \times 10^{-4} \text{ m}^3$, 16.52 kg/m^3 , 8.10%, 39.28 kN/m^2 , 60s, 10s and 60s is formed by mixing 1.25 kg polyol, 5mg silicon oil and 10g calcium carbonate (CaCO_3). Likewise, by mixing 1.2 kg polyol, 4mg silicon oil and 8g CaCO_3 , a rigid PUF with 0.005kg, $3.0861 \times 10^{-4} \text{ m}^3$, 16.2 kg/m^3 , 8.15%, 40.72 kN/m^2 , 50s, 15 cm and 58s key, physical and mechanical property as respectively listed under the flexible PUF formulation is produced. Both foams were produced using equal amounts of toluene diisocyanate, water, stannous octoate and methylene chloride, resulting in PUF that can be used in insulation, cushioning and construction support applications based on their characteristic height, density, tensile strength and compressive strength. As the surfactant, neem seed oil's potential in the synthesis of PUF cannot be overemphasized. The study of the kinetics of PUF production is limited and should trigger the adoption of biobased surfactants for industrial applications in the future.
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1. Introduction

Neem essential oil surfactant can be applied in the textile and petroleum industry [1, 2], including the production of polyurethane foam (PUF) [3]. PUF synthesis requires additives like stabilizers, blowing agents, foaming agents and surfactants. Rai, et al. [4] describe surfactants as chemical compounds that reduce surface or interfacial tensions of solvents or water when in contact with them. Petrochemicals are

still the chief precursors in which polyurethanes (PUs) are derived [5]. Dhaliwal [6] mentioned that Otto Bayer and his coworkers discovered PUF in 1937. Research is growing on using bio-based materials for PU production due to their appropriate flexibility, low cost, low toxicity, functionality, biocompatibility, biodegradability, unique combination of chemical structure, elasticity and abundance (sustainability) [7-9]. Excellent physical, chemical and mechanical properties of PU makes them an excellent material for multi-purpose application [7]. These applications includes films, coatings, elastomers, sealants and composite and polymer foams [10-14]. Neam, castor [15, 16], soya bean [17, 18], rapeseed [19], Lesquerella [20], *Jatropha curcas* [21], and orange [22, 23] seed oils had been used to synthesize PUF at normal pressure and temperature [24]. PFU with improved properties had also been achieved using modified rosin [25], epoxy vegetable oil [7, 26], alcohol-liquefied rice husk [27] and sugarcane fermentation by-products [28]. The synthesis often results in either flexible or rigid PUF [29, 30].

Flexible and rigid PUFs can be differentiated based on their chemical composition, density, mechanical properties, applications, thermal insulation, and manufacturing processes. Flexible PUF has low density, low flammability resistance, limited thermal insulation properties, and open cell structures with interconnected cells responsible for their flexibility, resilience, and compression [31]. It is chiefly produced by reacting polyols with diisocyanate and other additives using continuous or slab-stock foam manufacturing processes. On the other hand, the rigid type has a higher density than flexible PUF and provides excellent thermal insulation due to its closed-cell structures, contributing to its rigidity, low thermal conductivity [32] and structural support. It is formed by reacting polyols with diisocyanate and blowing agents (e.g., cyclopentane, water, hydrofluorocarbon, hydrofluoroethers) via a variety of manufacturing processes, including spray foam, pour-in-place, and panel production methods. Flexible PUFs are mainly used in comfort and cushioning applications, including furniture cushions, mattresses, pillows, upholstery and automotive seating [7, 25, 33]. Rigid PUFs are used for insulation and structural purposes, including insulation panels, doors, window frames, pipe insulation, aircraft insulation, refrigeration units and construction materials [6, 7, 34]. Castor oil is the most utilized essential oil as a surfactant in the production of PUF. Few studies demonstrate the use of neem seed oil as a surfactant. Hence, this study synthesizes rigid and flexible PUFs using a suitable formulation and analyses their physical and mechanical properties for several domestic and industrial applications. The study hopes to diversify PUF's chief precursor away from petrochemicals to achieve a favourable economic advantage.

2. Literature Review

Incorporating neem seed oil as a surfactant in the production of polyurethane foams presents several advantages and possibilities for enhancing the properties of the foams while promoting sustainability. Neem seed oil contains triglycerides and free fatty acids, which can act as surfactants. These compounds help

stabilize the foam structure by reducing surface tension, thus promoting uniform cell formation and enhancing the foam's overall properties. Including neem seed oil can improve the flexibility and rigidity of polyurethane foams. The oil's chemical composition helps balance soft and hard segments in the polymer matrix, leading to foams with desirable mechanical strength and flexibility. Neem oil and some nanoparticles are known for their antimicrobial properties [35]. Incorporating it into polyurethane foams can impart resistance to microbial growth, making them suitable for healthcare applications and environments requiring stringent hygiene standards. Due to their antimicrobial properties, neem seed oil-based polyurethane foams are suitable for medical devices, wound dressings, and other healthcare products, including insect repellent [36, 37]. The bio-based nature of neem oil aligns with the increasing demand for biocompatible and environmentally friendly materials in the medical field.

However, a literature search has shown that neem seed oil is less harnessed to demonstrate its potential for the above applications. Domingos manufactured PUFs from liquefied orange peel wastes. Liao, et al. [16] examined the influence of neem oil glyceride on the structure of PUF. Epoxidized orange seed oil has been used to produce PUF by Olu-Arotiowa, et al. [23]. Unmodified orange waste or orange seed oil was less utilized to manufacture PUF, based on the extent of this study investigation. So far, only Kuranchie, et al. [37] were reported to have purely used neem seed oil in their work, without considering the mechanical properties of the end-product. As such, using fresh neem seed oil as an important formulation ingredient of PUF production will significantly contribute to the existing study. Neem seed oil production typically emits fewer greenhouse gases than petrochemical processes due to its biogenic carbon content. Cultivation of neem trees can act as a carbon sink, offsetting some emissions. Neem oil is a renewable resource, whereas petrochemical surfactants deplete non-renewable fossil fuels. Energy use in neem oil extraction is generally lower than the energy-intensive processes of refining crude oil. Petrochemical surfactants may cause higher levels of ecotoxicity due to chemical runoff and spills. This study did not focus on the variability in neem oil production efficiency and yields, as well as the potential land use changes and competition with food crops if not managed sustainably – which present a major setback now. The variability in the composition of neem oil is due to factors like geographical origin and extraction methods, which can affect the consistency of the final product. A look at biomass blend utilization instead of single ones is needed to augment scarce ones in the future [38]. Whether for rigid or flexible types of foam, their properties have been improved severally for diverse or specific industrial applications [38, 39].

3. Materials and Method

3.1. Synthesis Materials

Equipment/instruments used during foam preparation in this study are an electronic laboratory weighing balance, meter rule, beaker, stirring rod, plastic container of various sizes, stopwatch and disposable

plastic cups;. In contrast, the materials used for foam preparation were polyol, toluene diisocyanate (TDI), neem oil stannous octoate, amine, silicon oil, methylene chloride, water and calcium carbonate (CaCO₃).

3.2. Foam Synthesis

About 1.25 kg of polyol and 10 mL of neem and silicon oil were mixed and poured into a disposable plastic container. It was mixed with 48 mL of water and vigorously stirred for 10 min. Next, 4 mL of stannous octoate and 500g of TDI were added to the mixture in step 2 and stirred thoroughly for 2-5 min. The mixture was then poured into a plastic mould. Immediately, the creaming was observed, simultaneously tailing with the rising of foam. The mixture was allowed to attain full rise and was kept curing for about 5 hours at room (or moulding) temperature. Two different formulations named Formulation A and Formulation B, whose constituent ingredients are shown in Table 1, were used to produce foam with distinct features.

Table 1. Formulation A and Formulation B Foam Produced Using Different Ingredients

Materials	Measurement	
	Formulation A	Formulation B
Polyol	1.25 kg	1.20 kg
Toluene diisocyanate	500 g	500 g
Neem seed oil	5 mg	3.5 mg
Silicon oil	5 mg	4 mg
Water	48 mL	48 mL
Stannous octoate	4 mL	4 mL
Methylene chloride	5.5 mL	5.5 mL
Calcium carbonate	10 g	8 g

Distinctively, Waziri, et al. [40] varied TDI concentration only for five formulations they experimented, Gimba, et al. [41] produced flexible PUF from 3 formulations differentiated by the ratios of olive oil surfactant to silicone oil used, while Usman, et al. [42] sought optimum based on seven heterogenous formulations using CaCO₃ as filler. In this work, the above materials were selected based on their unique role in forming foam [43], as described below. Polyol serves as one of the main components in the formation of PUF. It reacts with the TDI to form the PU polymer [13], contributing to the foam's flexible long segment structure and properties. Neem oil was used as a surfactant (stabilizer) in the production of PUF. Silicon oil is another surfactant used in foam production. It also stabilizes the foam structure and affects the properties of the final foam. Water reacts with TDI to produce CO₂, which creates the foaming action and contributes to the expansion of the foam [7]. Stannous acts as a catalyst in the reaction between the polyol and TDI, promoting the formation of the PU polymer. Methylene chloride was used as a blowing agent to help create the cellular structure of the foam, almost based on Stone, et al. [44] method. CaCO₃ was used as a filler to modify the properties of the foam, such as density, stiffness and tensile strength. Optimal mixing amounts

for plenty of ingredients can be a great challenge and may be influenced by many factors. To address this, Ogunleye and Oyawale [45] developed a linear programming scheme for selecting the optimal raw material mix for flexible PUF.

3.3. Density and Compressive Strength Measurements

In foam production, foam block height, length and width were measured to obtain the dimensions of the foam produced. The volume was then computed using Equation 1.

$$\text{Volume} = \text{Width} \times \text{Height} \times \text{Length} \quad (1)$$

The density of the foam samples was first determined by finding the weight (mass) of a regular-shaped foam block using an electronic weighing balance, and subsequently, the volumes obtained were plugged into Equation 2 to determine their density per ASTM D 1622 method [46, 47].

$$\text{Density} = \frac{\text{Mass of Foam (kg)}}{\text{Volume of Foam (m}^3\text{)}} \quad (2)$$

In some studies, a pycnometer was used to determine the neem oil density [2], and the method was described in DIN 53479 [48]. A compression test was carried out when the foam samples were in between two flat metals and subsequently compressed to 50% of their original size, as described by Abdullah, et al. [49]. It was allowed for 6 hours, and later, it was allowed to be recovered for 30 minutes. Next, the compressed foam samples were measured to determine the % change in original size and thickness. Essentially, the compression strength of the foams was evaluated using Equation 3.

$$\text{Compression (\%)} = \frac{(\text{Original Height}) - (\text{Final Height})}{(\text{Original Height})} \times 100 \quad (3)$$

This study conducted various tests according to ASTM D3574 standard [50, 51] and as described in Shivakumar, et al. [52].

3.4. Process Parameter Measurements

Cream time was obtained when a fixed amount of foam mixture was prepared, and the time taken for the mixture to reach a creamy consistency was recorded. To determine the foam rise, a fixed amount of foam mixture was poured into a container, and the vertical expansion of the foam was measured using a ruler. However, there is now a FOAM Software Package capable of simulating PUF expansion processes [48], which may be an answer to Powers [53] 's previous research goals and recommendations. In the case of free rising time, a fixed amount of foam mixture was poured into a container, and the time taken for the foam to rise freely without external constraints was recorded. The foam's cell structure was observed and recorded for cell opening observation. This process was repeated for both Foam A and B samples using a cup test utilized in Kraitape and Thongpin [46]. All measurements were taken using standard laboratory equipment and techniques.

4. Results and Discussion

4.1. Produced Foam

Figure 1a is the produced flexible PUF using Sample A composition formulation while Figure 2 is the produced rigid PUF using another composition formulation (Sample B), as earlier reported in Table 1.



Figure 1. (a) Flexible Formulation A and (b) Rigid Formulation B Foam

From the two different foam formulations (Foam A and B samples) showcased in Table 1, it is observed that varying the amount of TDI controls the hardness of the foam. Increasing the amount correspondingly increases its density, as observed in those samples. Both density and hardness increase as the amount of polyol increases [54]. An increase in dimethylamine decreased the density of foam samples, as evidenced in Table 3. However, a decrease in dimethylamine initiates the reaction between TDI and water to increase the production of CO₂ gas during foaming, which is responsible for the increase in the volume of the sample.

4.2. Physical and Mechanical Properties

Table 2 shows the dimensions of Foam A and B samples. Table 3 shows the volume, mass, and densities of the produced foams. Tables 4 and 5 show the strength properties of Foam A and B formulations and other properties.

Table 2. Foam Dimensions

Dimension (m)	A	B
Height	0.1300	0.1270
Length	0.0995	0.0900
Breath	0.0220	0.0270

Table 3. Volume and Densities of Foam A and B

Formulations	Volume $\times 10^{-4}$ (m ³)	Mass (kg)	Density (kg/m ³)
A	2.8457	0.0047	16.52
B	3.0861	0.0050	16.20

Table 4. Strength Properties of Foam A and B Samples

Samples	Tensile Strength (kN/m ²)	Compression (%)
A	39.28	8.10
B	40.72	8.15

Table 5. Vital Metrics of the Respective Foams Manufacture

Parameter	A	B
Cream time (s)	60	50
Foam Rise (cm)	10	15
Free Rising Time (s)	60	58
Cell Opening	α	δ

The effect of these physical, chemical, and mechanical properties can be discussed, and suggestions can be made on where they best fit to be applied in the practical world.

4.3. Foam Selection Based on Dimensions and Density

The height, length, and width of foams, as indicated in Table 2, can indeed be significant in various applications. For example, the dimensions are crucial for ensuring proper fit and functionality in applications where the foam is used as a cushioning or insulating material [25]. In the furniture industry, foam cushions must fit precisely within the frames to provide comfort and support. Foam materials are commonly used for packaging and shipping to protect fragile items. Dimensions of the foam pieces need to align with the dimensions of the packaged items to provide adequate protection during transit. In construction and building applications, foam materials are used for insulation, soundproofing, and structural support [6,35], in which the dimensions of the foam sheets or blocks need to match the specific requirements of the construction project. Foam height ranging from 3.5-6.0 cm obtained by Onyema [55] using *Securidaca longepedunculata* as surfactant is very low compared to 13 cm (0.13 m) and 12.7 cm (0.127 m) Foam A and B heights, obtained in this study.

All the ingredients contributed to the foams' mass, volume and density. In a study carried out by Liao, et al. [16], densities of PUF ranged from 49.7-116.2 kg/m³ above those recorded for Foam A and B. Ekkaphan, et al. [56] measured a density of 40-45 kg/m³ for rigid PUF. In this study (Table 3), Formulation B has a lower density (16.20 kg/m³) compared to Formulation A (16.52 kg/m³), indicating that it is less dense. These densities are within 12 kg/m³ minimum and 40 kg/m³ maximum ISO values [57]. Formulation B also has a slightly higher mass (0.0050 kg) and volume (3.0861×10^{-4} m³) compared to Formulation A (0.0047 kg and 2.846×10^{-4} m³, respectively). Therefore, in terms of density, mass, and volume, Formulation B is the better formulation and is rigid. Therefore, Foam B may be recommended for the furniture

industry and packaging and shipping applications due to its higher volume and lower density. Also, Foam B, with its higher volume, may be more suitable for custom-cut foam pieces where flexibility in shaping and sizing is required to meet specific project needs. For specific requirements, foams A and B could be suitable for construction and building applications. Foam A's slightly higher density may offer advantages in structural support, while Foam B's lower density and higher volume may benefit insulation [58] and soundproofing.

Flexible PUF densities can vary widely depending on the application. Examples are low-density foam with 8-32 kg/m³ (0.5-2.0 lb/ft³) density used in furniture cushions, medium-density foam with 32-64 kg/m³ (2-4 lb/ft³) density, commonly used in mattresses and high-density foam with ≥ 32 kg/m³ (4 lb/ft³) density used for speciality applications, such as medical mattresses. On the other hand, common densities for rigid PUF range from about 32-160 kg/m³ (2-10 lb/ft³). Chen also studied the compressive behaviour of various density PUFs.

4.4. Tensile and Compressive Strength Property

Tensile strength measures the foam's ability to withstand stretching or pulling forces, while compressive strength assesses its capacity to resist squeezing or crushing. Foam A exhibits a tensile strength of 39.28 kN/m² and a compressive strength of 8.10%, while Foam B demonstrates a tensile strength of 40.72 kN/m² and a compressive strength of 8.15%, as shown in Table 4. These values indicate that Foam B has slightly higher tensile and compressive strength than Foam A. The higher tensile strength of Foam B suggests that it may be more resistant to stretching forces, making it potentially suitable for applications where tensile forces are a concern, such as in cushioning or support applications. Similarly, the higher compressive strength of Foam B indicates its ability to withstand greater crushing forces, which could be advantageous in applications where the foam is subjected to pressure or weight [56, 57], such as in structural or load-bearing uses. However, there is still room for improving the tensile strength obtained in this study, given that it is lower than the 70 and 240 kN/m² minimum and maximum ISO values reported by Oyetunji and Hammed [57]. Because of that, other materials with filling properties such as dolomite [66], talc, eggshell, kaolin (clay), wood flour, cellulose fibres, alumina trihydrate, barium sulphate, mica, rice husk ash, bamboo fibres, metal powders (e.g., Al, Fe), carbon black, silica, mica, glass microspheres and wollastonite may be tested to find potential alternative. Already, Victor, et al. [59] and Eberonwu, et al. [60] reported a better filling property of chicken eggshells compared to the conventional use of CaCO₃, and Aghvami-Panah, et al. [61] examined carbon black. Particle sizes of the filler may also improve the mechanical properties of the PUF [54].

4.5. Key Process Parameters for PUF Production

Parameters listed in Table 5 offer valuable insights into the performance and behaviour of the foams, shedding light on various characteristics beyond tensile and compressive strength. Cream time indicates

the time the foam takes to reach a creamy consistency during the production process or the time interval in which the mixture begins to expand [53]. Foam B exhibits a shorter cream time (50s) than Foam A (60s), suggesting that Foam B may have a faster reaction time and potentially more efficient production characteristics. Foam rise measurement reflects the vertical expansion of the foam during production. In Kollia, et al. [47], it is mentioned that as the water content is increased, gel time, foam rise and density of the PUF decreases. Foam B demonstrates a higher foam rise (15 cm) than Foam A (10 cm), indicating that Foam B expands to a greater extent, potentially offering advantages in filling molds or achieving specific shapes. Free rising time represents the time taken for the foam to rise freely without external constraints [34]. Foam A and B exhibit relatively similar free rising times, with Foam B showing a slightly shorter duration (58s) than Foam A (60s). Gimba et al. [41] also realized that the " α " symbol in the cell opening column suggests that Foam A has a fine or open-cell structure. Open-cell foams typically offer enhanced breathability and flexibility, making them suitable for applications where airflow and softness are desired [62]. Rigid PUF has a closed-cell structure, which means its cells are not interconnected. Hence, the case with Formulation B represented by " δ " implies rigidity or a closed-cell structure. This closed-cell structure contributes to its excellent insulation properties by reducing the flow of air and moisture through the material.

5. Conclusion

Beyond mere formulation of a flexible "A" and rigid PUF "B", this study further suggests areas in practical application they all fit into based on their physical and mechanical properties. Formulation A produces flexible PUF that best suits building and construction structural support applications due to its lower volume of $2.846 \times 10^{-4} \text{ m}^3$ and high density. Formulation B results in rigid PUF with 16.20 kg/m^3 density that can go into insulation, furniture, packaging and shipping applications due to its low density and high volume, also being able to resist stretching due to its higher tensile strength of 40.72 kN/m^2 than A. The selected neem seed oil surfactant's ability to produce two classes of PUF in this study is enough to re-orient PUF manufacturers to harness it further as the chief ingredient in rigid and flexible PUF formulations. There are also numerous raw materials with great potential to serve as blowing agents and fillers, apart from the conventional use of water and CaCO_3 . Finding the optimal combination for each surfactant, filler, blowing agent, or all ingredients used will convince manufacturers of the cost-effectiveness of the related studies' outcome. Already, sustainability and environmental friendliness are issues with using petrochemicals for PUF production that have been partly addressed by neem seed oil. A beautiful, attractive, flame-retardant foam can be realized using colourants and polybrominated diphenyl ether additives.

Declaration of Competing Interest The authors declare they have no known competing interests.

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